



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ :

C03C 15/00, C03B 37/00

A1

(11) International Publication Number:

WO 98/14409

(43) International Publication Date:

9 April 1998 (09.04.98)

(21) International Application Number: PCT/US97/15895

(22) International Filing Date: 8 September 1997 (08.09.97)

(30) Priority Data:

60/027,256

30 September 1996 (30.09.96) US

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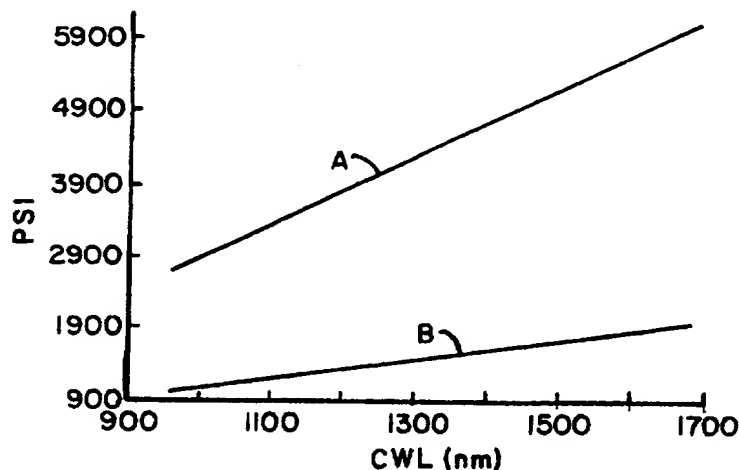
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(81) Designated States: JP, KR, US, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

Published

With international search report.

(54) Title: METHOD OF MAKING A POLARIZING GLASS



(57) Abstract

A method of producing a polarizing glass article that exhibits a broad band of high contrast polarizing properties in the infrared region of the radiation spectrum, that is phase-separated or exhibits photochromic properties based on silver, copper, or copper-cadmium halide crystals or a combination thereof precipitated in the glass and having a size in the range of 200-5000 Angströms, and that has a surface layer containing elongated silver, copper, or copper cadmium metal particles, or a mixture of same, the method comprising the step of thermally forming and precipitating large halide crystals by subjecting the glass article to a time-temperature cycle in which the temperature is at least 75 °C above the glass softening point. The figure is a graphical representation of stress levels in psi versus center wavelengths (CWLs) in nm, where center wavelength is defined as the center of a given range of polarizing capability. Specifically, the stress levels required to achieve a given center wavelength is compared for two heat treatment cycles. The upper line (A) represents data for the standard heat treatment of polarizing glasses; 710 °C for 4 hours. Line (B) represents data for the new high temperature heat treatment of 750 °C for 8 hours.

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METHOD OF MAKING A POLARIZING GLASS

FIELD OF THE INVENTION

5 Method of making a polarizing glass from a phase-separated glass containing silver, copper, or copper-cadmium halide crystals.

BACKGROUND OF THE INVENTION

10 It is known to induce the precipitation of silver, copper, or copper-cadmium halide crystals by heat treatment of glasses having compositions containing the indicated metal, and a halogen other than fluorine, in suitable amounts. The resulting glasses usually exhibit photochromic behavior, that is, they darken and fade in response to application and removal of short wavelength radiation. However, it is possible to
15 produce glasses that contain the indicated crystals, but are not photochromic.

 A birefringent effect can be generated in these crystal-containing glasses by stretching the glass within a certain viscosity range. The glass is placed under stress at a temperature above the glass strain point temperature. This elongates the glass, and thereby elongates and orients the crystals. The elongated article is then exposed to a
20 reducing atmosphere at a temperature above 250°C, but not over 25°C above the glass annealing point. This develops a surface layer in which at least a portion of the halide crystals are reduced to elemental metal. The elongated elemental crystals provide an array of electric dipoles which preferentially interact with the electric field vector of incident light. This provides a method to polarize transmitted light waves.

The production of a polarizing glass, then, involves, broadly, these four steps:

1. Melting a glass batch containing a source of silver, copper, or copper-cadmium and a halogen other than fluorine, and forming a body from the melt,
2. Heat treating the glass body at a temperature above the glass strain point
5 to generate halide crystals having a size in the range of 200-5000 Å,
3. Stressing the crystal-containing glass body at a temperature above the glass strain point to elongate the body and thereby elongate and orient the crystals, and
4. Exposing the elongated body to a reducing atmosphere at a temperature
10 above 250°C to develop a reduced surface layer on the body that contains metal particles with an aspect ratio of at least 2:1.

Practice of this method produces glass articles displaying excellent polarizing properties over the infrared portion of the radiation spectrum, preferably within the region of 600-2000 nm (6000-20,000 Å).

15 The growth of halide particles cannot occur at temperatures below the strain point of the glass because the viscosity of the glass is too high. Therefore, temperatures above the annealing point are preferred for crystal precipitation. Where physical support is provided for the glass body, temperatures up to 50°C above the softening point of the glass can be employed.

20 Experience has demonstrated that the halide crystals should have a diameter of at least about 200 Å in order to assume upon elongation, an aspect ratio of at least 5:1. When reduction to elemental particles occurs, the particles having an aspect ratio of at least 5:1 will display an aspect ratio greater than 2:1. This places the long wavelength peak at least near the edge of the infrared region of the radiation spectrum, while avoiding serious breakage problems during the subsequent elongation step. At the other
25 extreme, the diameter of the initial halide particles should not exceed about 5000 Å. This precludes the development of significant haze in the glass accompanied with a decreased dichroic ratio resulting from radiation scattering.

30 The dichroic ratio is a measure of the polarizing capability of a glass. It is defined as the ratio existing between the absorption of radiation parallel to the direction of elongation and the absorption of radiation perpendicular to the direction of elongation. To attain an adequate ratio, the aspect ratio of the elongated halide crystals

must be at least 5:1 so that the reduced metal particles have an aspect ratio of at least 2:1.

Crystals having a small diameter demand very high elongation stresses to develop a necessary aspect ratio. Also, the likelihood of glass body breakage during a stretching-type elongation process is directly proportional to the surface area of the body under stress. This creates a very practical limitation as to the level of stress that can be applied to a glass sheet, or other body of significant mass. In general, a stress level of a few thousand psi has been deemed to comprise a practical limit. Stress levels above 3000 psi are customarily used.

Firing of the elongated body in a reducing atmosphere is undertaken at temperatures above 250°C, but no higher than 25°C above the annealing point of the glass. Preferably, the firing temperature is somewhat below the annealing point of the glass to prevent any proclivity of the particles to respheroidize.

Thus, as the temperature of the reduction step, or of any other heat treatment subsequent to the elongation step, is increased, there is a tendency for the elongated particles to return to their original state, or to break into small particles. This tendency had been termed respheroidizing. This tendency places a serious limitation on the temperature at which any such subsequent heat treatment may be undertaken.

One of the key measures of the effectiveness of a polarizing glass body is its contrast ratio, or, as referred to in the art, contrast. Contrast comprises the ratio of the amount of radiation transmitted with its plane of polarization perpendicular to the elongation axis to the amount of radiation transmitted with its plane of polarization parallel to the elongation axis. In general, the greater the contrast, the more useful, and valuable, the polarizing body. Another important feature of a polarizing body is the bandwidth over which the body is effective. This property takes into consideration not only the degree of contrast, but the portion of the spectrum within which the contrast is sufficiently high to be useful.

The level of contrast attainable in a polarizing glass body is dependent upon the amount of reduction occurring during the step of firing in a reducing atmosphere.

Typically, the greater the extent of reduction the greater the level of contrast. Thus, the

degree of contrast can be increased by employing either higher temperatures, longer times, or higher pressures of reducing gas species for reduction.

That practice is limited, however, by the tendency of the metal halide particles to respheriodize. That tendency is also enhanced by higher temperatures and longer times of firing. Respheriodization can result in a decrease in contrast and/or a narrowing of the peak absorption band, or a shifting of the peak absorption band in the direction of shorter wavelengths. To illustrate, a process for preparing polarizing glass articles in accordance with prior knowledge has utilized firing in a hydrogen atmosphere for 4 hours at 425°C. When the firing time was extended to 7 hours, the contrast was increased somewhat, but with a concurrent reduction in the bandwidth of high contrast.

United States Patent No. 4,908,054 (Jones et al.) proposes a method of producing a polarizing glass body that obviates the effect of respheriodization during a heat treatment such as the reduction step. This method conducts the thermal reducing treatment under a pressure of at least twice atmospheric pressure. The effect of the pressure is to inhibit respheriodization and to produce a polarizing glass article that exhibits a relatively broad range of high contrast polarizing properties in the infrared region. This expedient is not required in the present invention, but may be employed.

It is a purpose of the present invention to provide a glass article that has excellent polarizing properties over a wide range of the radiation spectrum. Another purpose is to accomplish this with or without employing the Jones et al. expedient of enhanced pressure. A further purpose is to provide a phase-separated glass that is elongated at a relatively low stress level. A still further purpose is to produce a polarizing glass article having a relatively flat contrast absorption curve over a wide wavelength band.

SUMMARY OF THE INVENTION

The invention resides in a method of producing a glass article that exhibits a broad band of high contrast polarizing properties in the infrared region of the radiation spectrum, that is phase-separated, or exhibits photochromic properties, based on silver, copper, or copper-cadmium halide crystals precipitated in the glass within a size range of

200-5000 Å, and that contains elongated silver, copper, or copper-cadmium metal particles, the method comprising thermally forming and precipitating large halide crystals in the glass article by subjecting it to a time-temperature cycle in which the temperature is at least 75°C above the glass softening point, preferably greater and the time is
5 sufficient to form the crystals, preferably over one hour, and elongating the glass article at a temperature between the strain point and the softening point of the glass.

The invention further resides in a method for making a glass article exhibiting a relatively broad band of high contrast polarizing properties in the infrared region of the radiation spectrum from glasses which are phase-separable, or exhibit photochromic
10 properties, through the presence of silver, copper, or copper-cadmium halide crystals, the method comprising the steps of:

(a) melting a batch for a glass containing a source of silver, copper, or copper-cadmium and at least one halogen other than fluorine or a combination thereof,

(b) cooling and shaping the melt into a glass article of a desired
15 configuration,

(c) subjecting the glass article to a temperature at least 75°C above the softening point of the glass to generate and precipitate silver, copper, or copper-cadmium crystals in the glass, the crystals ranging in size between about 200 and 5000 Å,

(d) elongating the glass article under stress not over about 3000 psi at a
20 temperature above the strain point of the glass to provide the crystals with an elongated aspect ratio of at least 5:1 and aligned in the direction of the stress, and,

(e) exposing the elongated glass article to a reducing atmosphere at a temperature above about 250°C, but no higher than about 25°C above the annealing
25 point of the glass for a period of time sufficient to develop a reduced surface layer on the glass article wherein at least a portion of the elongated halide crystals are reduced to elemental silver, copper, or copper-cadmium particles having aspect ratios greater than 2:1 which are deposited in and/or upon said elongated crystals, whereby the glass article exhibits a relatively broad range of high contrast polarizing properties in the infrared
30 region of the radiation spectrum.

BRIEF DESCRIPTION OF THE DRAWINGS

FIGURE 1 is a graphical representation comparing the stress levels required in accordance with the present invention, as compared to prior practice, to achieve a given center wavelength in a polarizing glass.

FIGURE 2 is a graphical representation of a typical contrast ratio curve obtained with the present invention as compared to a similar curve obtained with prior practice.

FIGURE 3 is a graphical representation comparing maximum transmission values for a polarizing article produced in accordance with the present invention as compared to one produced in accordance with prior practice.

PRIOR ART

Prior literature of possible interest is listed and described in an attached document.

DESCRIPTION OF THE INVENTION

The present invention adopts, and improves on, the known method of producing a polarizing glass body. Basically, it embodies the steps of melting, and forming an article from, a glass containing a source of silver, copper, or copper-cadmium and a halogen or combination thereof other than fluorine. The article is cooled and then heat treated to form and precipitate halide crystals of silver, copper, or copper-cadmium. The article is then heated and subjected to stress to elongate the halide crystals. The glass is then subjected to a thermal reduction step, preferably in a hydrogen atmosphere, to reduce a portion of the silver or copper halide crystals in a surface layer on the article to elongated metal particles.

The glass employed may be any glass that can be phase-separated to form silver, copper, or copper-cadmium crystals in the glass. Such glasses are disclosed, for example, in United States Patent Nos. 4,190,451 (Hares et al.) and 3,325,299 (Araujo) disclosing photochromic glasses and 5,281,562 (Araujo et al.) disclosing non-

photochromic glasses. Each of these patents is incorporated by reference, particularly for its teaching of glass composition ranges and their production. Preferred glasses are those disclosed in the Hares et al. patent. The present invention is concerned with a modification of the step in which the glass is heat treated to form and precipitate the halide crystal phase. It has been suggested in the literature that this step may be carried out at any temperature in the range of 500-900°C. However, the Jones et al. patent specifies what has been accepted as good practice. The temperature is specified to be

above the glass strain point, but not over 75°C above the glass softening point. Temperatures considerably below the 75°C over the glass softening point are disclosed in examples. The time is stated to be a sufficient time to generate halide crystals.

The maximum temperature is dependent on the viscosity characteristics of the glass. In general, the temperature should not be so high that the glass becomes undesirably soft, a viscosity point of about 10^5 poises being a practical limit.

It has now been found that new and unexpected benefits are achieved by a modified heat treatment to produce the halide crystals in the glass. In particular, the glass is heat treated at a temperature of at least 75° above the softening point, and for time sufficient to develop the crystals, usually at least greater than about an hour.

These higher heat treatment temperatures have produced glass blanks with larger, and a wider variety of, crystal sizes. The increase in metal halide crystal size permits elongating the glass at a much lower stress level, preferably not over 3000 psi. This improves the operation and lessens the chance for breakage during the stretching process. The dramatic decrease in required pulling forces is shown in FIGURE 1.

FIGURE 1 is a graphical representation in which stress levels are plotted in psi on the vertical axis and center wavelengths (CWLs) in nm are plotted on the horizontal axis. The center wavelength is that wavelength at the center or peak of a given range of polarizing capability. Treatment conditions will be targeted to the desired wavelength for a particular application.

In FIGURE 1, the stress levels required to achieve a given center wavelength are compared for two heat treatment cycles. The upper line A represents data from a standard heat treatment at a temperature of 710°C for 4 hours. This cycle is typical of the cycles employed for several commercial polarizing glass products. It will be

observed that a product having a center wavelength at 1310 nm requires a stress level around 3400 psi. The lower line B presents data for a new high temperature heat treatment of 750°C for 8 hours. This cycle is in accordance with the present invention. In this case, the stress level needed to achieve 1310 nm center wavelength is only about 1600 psi.

A second benefit achieved by the new heat treatment process is a wider dispersion of crystal sizes. Thus, a flatter contrast absorption curve over a wider wavelength band is obtained than that normally obtained with the prior lower temperature heat treatment process. This relationship is shown graphically in FIGURE 2.

FIGURE 2 is a graphical representation in which Contrast Ratio is plotted on the vertical axis and wavelength is plotted in nm on the horizontal axis. The upper curve C shows the relationship of contrast ratio to wavelength for a commercial polarizing glass article. This article was processed on a schedule of 710°C for 4 hours to precipitate halide crystals. The lower curve D shows the same relationship for the same article produced from the same glass, but heat treated at 750°C for 8 hours to develop halide crystals in accordance with the method of the present invention. It is apparent that curve D is a broader, flatter curve.

An indirect measure of the size and extent of halide crystallization, the degree of optical transmittance and the extent of light scattering (haze) is used to monitor the effect of a particular heat treatment. This glass composition would normally fall into the range of 10-20% haze if treated at 50°C above the softening point. Treatments at approximately 90°C or greater than the softening point render haze readings which have reached 100%. In a similar way, a heat treatment at 50°C above the softening point yields a transmission (T_{\max}) of approximately 91 to 93% for this glass. A much higher heat treatment of 90°C above the softening point lowers T_{\max} to 84-88%. Still higher heat treatments continue to lower the transmission.

The penalty in lowered transmission for polarizing glass products made with the higher heat treatment process is small as long as the transmitting wavelength is high (longer wavelength). This effect is depicted in FIGURE 3 which compares the

wavelength dependence on transmission for the normal 710°C/4 hour treatment, upper curve E, to the new high temperature treatment of 750°C/8 hours, lower curve F.

FIGURE 3 is a graphical representation in which maximum transmission (T_{\max}) is plotted on the vertical axis in %. Wavelength is again plotted in nm on the horizontal axis. As shown, the difference in transmission loss becomes less than 10% at wavelengths above about 800 nm, and becomes insignificant above about 1100 nm.

The glass employed in making test pieces to obtain the data presented in the drawings has the following composition in % by weight as calculated from the batch on an oxide basis:

10	SiO ₂	56.3	ZrO ₂	5.0
	B ₂ O ₃	18.2	TiO ₂	2.3
	Al ₂ O ₃	6.2	Ag	0.24
	Na ₂ O	5.5	CuO	0.01
	Li ₂ O	1.8	Cl	0.16
15	K ₂ O	5.7	Br	0.16.

WE CLAIM:

1. A method of producing a polarizing glass article that exhibits a broad band of high contrast polarizing properties in the infrared region of the radiation spectrum, that is phase-separated or exhibits photochromic properties based on silver, copper, or copper-cadmium halide crystals precipitated in the glass and having a size in the range of 200-5000 Å, and that has a surface layer containing elongated silver, copper, or copper cadmium metal particles, the method comprising thermally forming and precipitating large halide crystals in the glass article by subjecting it to a time-temperature cycle in which the temperature is at least 75°C above the glass softening point, preferably greater and the time is sufficient to form the crystals, preferably over one hour, and elongating the glass article at a temperature between the strain point and the softening point of the glass.
2. The method of claim 1 which comprises subjecting the glass article to a time-temperature cycle in which the glass has a viscosity not less than 10^5 poises and the time is not over 20 hours.
3. The method of claim 1 which comprises the further step of subjecting the glass article containing the halide crystals to an elongating stress level sufficient to impart an aspect ratio of at least 5:1 to the crystals.
4. The method of claim 3 which comprises the further step of exposing the elongated glass article to a reducing atmosphere at a temperature over 250°C, but not over 25°C above the glass annealing point, to form silver, copper, or copper-cadmium metal particles in a surface layer on the article.
5. The method of claim 4 which comprises using a reducing gas at an elevated pressure.

6. A method of producing a polarizing glass article that exhibits a broad band of high contrast polarizing properties in the infrared region of the radiation spectrum, that is phase-separated or exhibits photochromic properties based on silver, copper, or copper-cadmium halide crystals precipitated in the glass and having a size in the range of 200-5000 Å, and that has a surface layer containing elongated silver, copper, or copper cadmium metal particles, the method comprising the steps of

- (a) melting and forming an article from a glass containing a source of silver, copper, or copper-cadmium and a halogen other than fluorine, or a combination thereof,
- (b) thermally forming large halide crystals in the glass article by subjecting it to a time-temperature cycle in which the temperature is at least 75°C above the glass softening point for appropriate time,
- (c) applying a stress to the glass article while it is at a temperature above the glass strain point to elongate the article and the halide crystals therein,
- (d) exposing the elongated glass article to a reducing atmosphere at a temperature above about 250°C to reduce at least a portion of the halide crystal particles in a surface layer on the article to form elongated metal particles.

7. A method in accordance with claim 6 which comprises melting a glass containing a source of silver, and thermally forming large silver halide crystals in the glass.

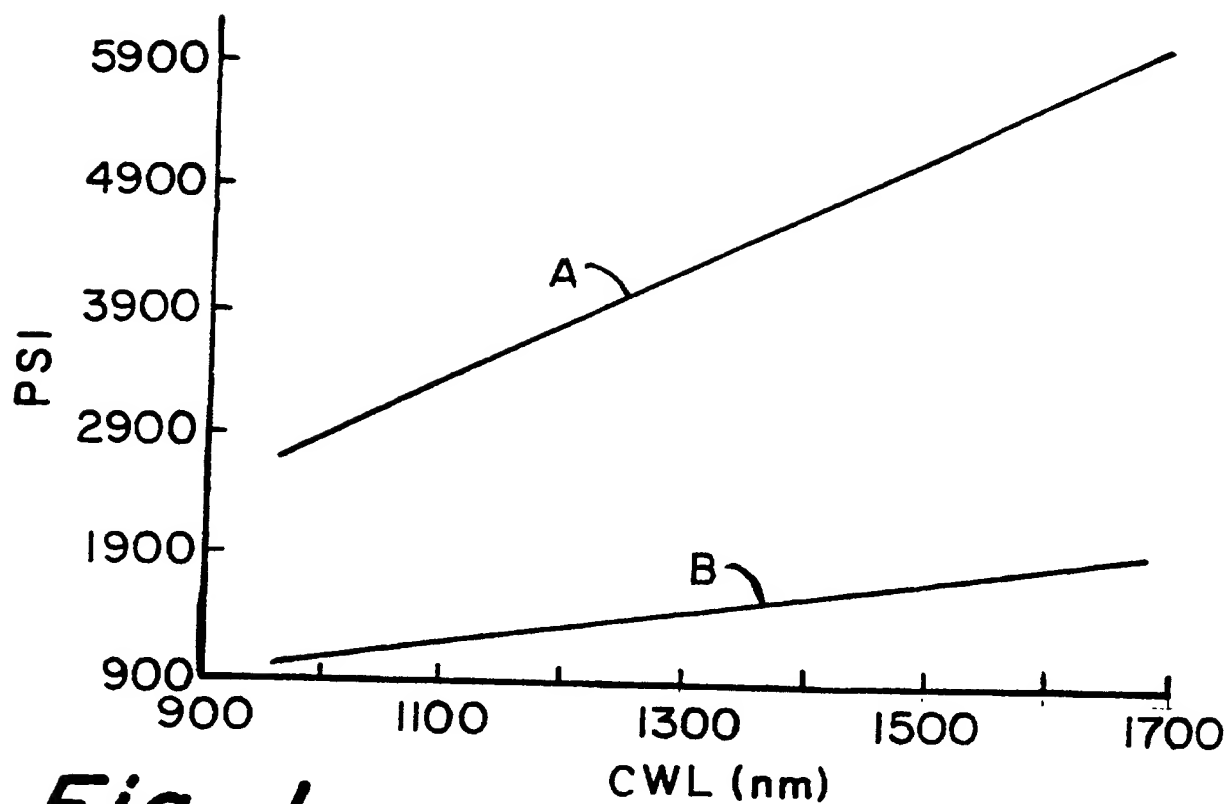
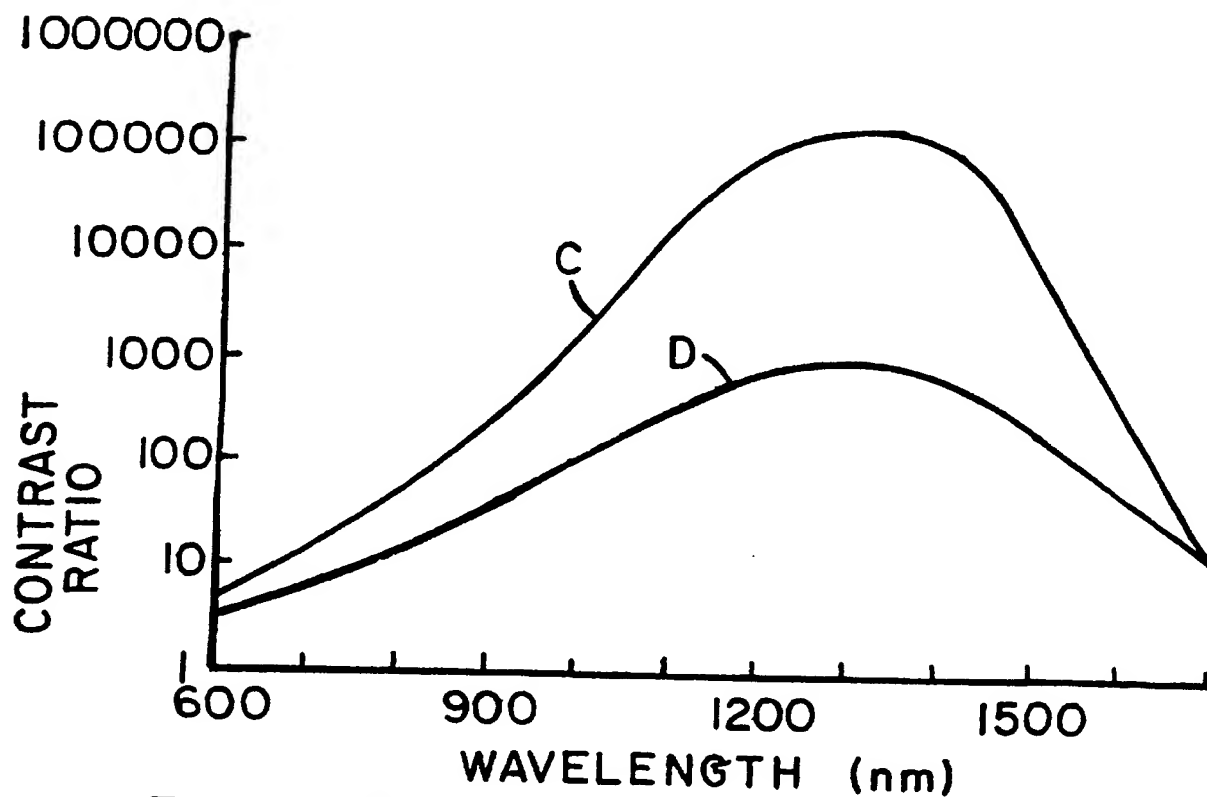
8. A method in accordance with claim 6 which comprises melting a glass containing a source of copper, and thermally forming large copper halide crystals in the glass.

9. A method in accordance with claim 6 which comprises forming large halide crystals by subjecting the glass to a time-temperature cycle in which the temperature is at least 75°C above the glass softening point and the time is 1-20 hours.

10. A method in accordance with claim 6 which comprises applying an elongating stress level to the crystal-containing article that is sufficient to impart an aspect ratio of at least 5:1 to the crystals.

11. A method in accordance with claim 10 wherein elongating stress level is not over about 3000 psi.

5 12. A method in accordance with claim 6 which comprises exposing the elongated glass article to a reducing atmosphere at a temperature over 250°C, but not over 25°C above the glass annealing point to form silver, copper, or copper-cadmium metal particles in a surface layer on the glass article.

**Fig. 1****Fig. 2**

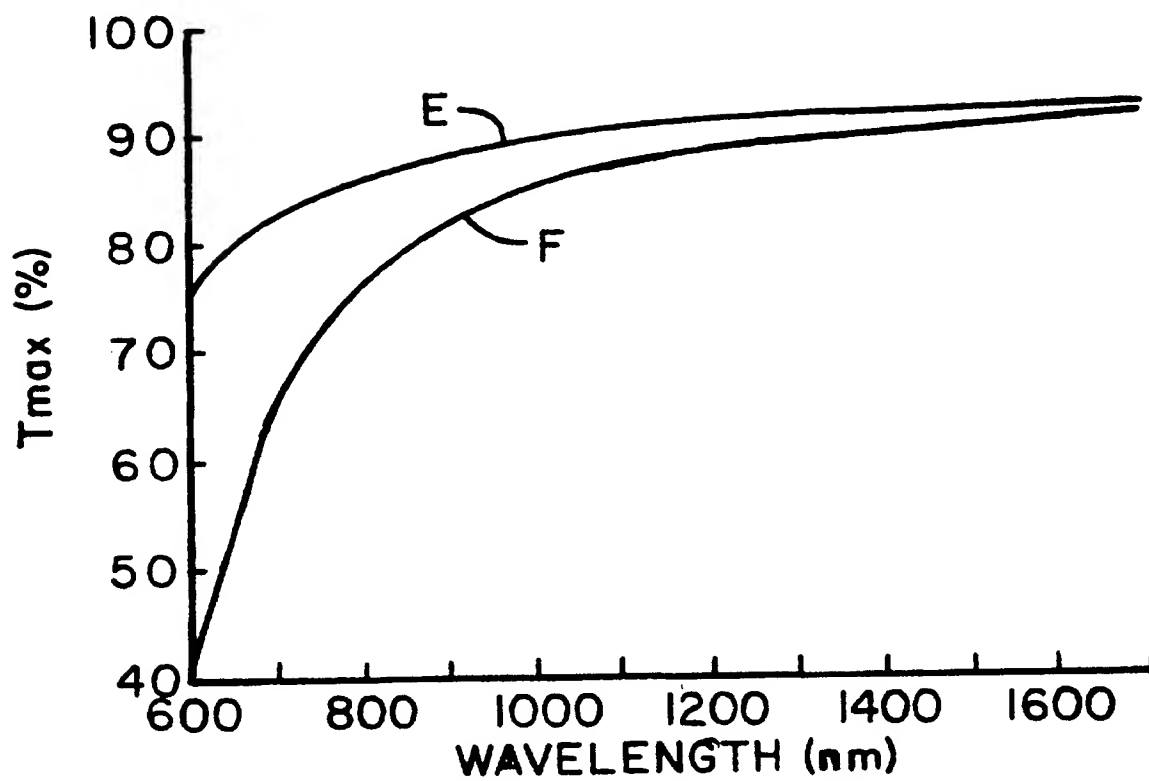


Fig. 3

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US97/15895

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : C03C 15/00; C03B 37/00

US CL : 65/30.1, 30.11, 32.1, 32.3, 33.1; 501/13

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 65/30.1, 30.11, 32.1, 32.3, 33.1; 501/13

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
APS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X --- Y	US 4,908,054 A (JONES et al) 13 MARCH 1990, abstract, col. 5, lines 40-50, claim 1.	1-7 ----- 8-12
Y	US 3,653,863 A (ARAUJO et al) 04 APRIL 1972, col. 5.	1-7
Y	US 4,405,672 A (ARAUJO et al.) 20 September 1983, abstract.	1,6,8
Y	US 4,190,451 A (HARES et al) 26 FEBRUARY 1980, abstract.	1,6,8

☐ Further documents are listed in the continuation of Box C.☐ See patent family annex.

* Special categories of cited documents:	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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Date of the actual completion of the international search

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